

TECHNICAL REPORT

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# EFFECT OF MOISTURE ON THE STABILITY OF FLEXIBLY-PACKAGED SYNTHETIC DRY VINEGAR

by

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NATICK LABORATORIES  
Natick, Massachusetts 01760



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13. ABSTRACT A free flowing dry vinegar capable of withstanding high temperature storage resulted when a moisture-proof packaging material and in-package desiccation was used. Since the interchange of moisture between product and desiccant occurred at a slow rate, the level of desiccant was contingent on the moisture level to be extracted which was 0.2 percent out of total of 0.3 percent. The deterioration reaction which occurred at the higher moisture level was completely eliminated when the product moisture level was maintained at less than 0.1 percent.			

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Storage Stability	7		7		8	
Vinegar	7		7		9	
Dry	0		0		0	
Synthetic	0		0		0	
Desiccants			6		10	
Packaging Materials			6		10	
Flexible			0		0	

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### Foreword

The U.S. Army Natick Laboratories in cooperation with industry, has developed a flexibly-packaged synthetic dry vinegar. This new dry vinegar is a free-flowing, noncorrosive mixture with minimal odor whereas the old dry vinegar as specified in MIL-V-35017B is a nonflowing slurry with a pungent odor, limited stability, and requires careful handling.

However, the new vinegar has limitations. The product will deteriorate in texture and color when subjected to elevated storage temperatures (100°F). In addition, delamination of packaging materials occurs. The purpose of this study was to determine corrective measures to prevent this deterioration.

The authors wish to acknowledge the assistance of Messrs Earl Merwin and John Walsh, McCormick Company in providing materials and technical assistance in this project. Assistance of Mr. Jesse Hill, General Equipment and Packaging Laboratories, NLABS is also acknowledged.

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### Abstract

A free flowing dry vinegar capable of withstanding high temperature storage resulted when a moisture-proof packaging material and in-package desiccation were used. Deterioration reactions which occurred at higher moisture levels were completely eliminated when the product moisture level was maintained at less than 0.1 percent.

## Introduction

A new free flowing dry vinegar has been developed which when reconstituted has a sweet-tart taste and aroma similar to commercially available 50 grain vinegar. Burt (1969) reported that this new dry vinegar is equal to commercial vinegar in acceptability. The product is a modification of dry vinegar covered by U.S. Patent 2,696,441 which was issued to Kamieciek and Farrell (1955). Oser's patent (1924) was the first to cover the general area of dry vinegar.

The new dry vinegar has limitations, however, in that the texture and appearance are adversely affected when the product is stored at 100°F. or higher. It develops a pungent acetic acid odor and becomes a brownish slurry with subsequent delamination of the packaging material.

This investigation was conducted to determine whether the product deterioration is due to any of the following causes:

1. Moisture transfer through packaging materials.
2. Poorly sealed or leaking packages.
3. Interaction of ingredients with residual moisture (0.35%).

## Experimental Methods

Dry vinegar was made in accordance with Military specification MIL-V-0035017C, Type II, dated 6 Jul 71. This dry vinegar is a blend of malic acid, a sodium salt of acetic acid, caramel coloring, apple flavor and tricalcium phosphate. The proportion of malic acid to acetic acid salt is 50:50 by weight. The product is a homogenous free flowing mixture with a moisture content of not more than 0.35 percent.

In the first experiment, the product was packed in sealed mason jars and stored at varying temperatures: (72°F, 85°F, 90°F, 100°F, 110°F and 140°F) to determine the effect of temperature on product stability.

In the second experiment, the product was packaged in a moisture-proof, flexible, laminated package constructed from outside to inside of 0.005 inch polyester laminated to 0.0035 inch aluminum foil laminated to 0.003 inch polypropylene. To study the effects of moisture, some 3-ounce packages of product were packed with  $\frac{1}{4}$  oz. of bagged desiccant (calcium oxide) and some without the desiccant. Based on calculations using the formula in MIL-D-43266A, "Desiccants and Desiccation, Method of,"  $\frac{1}{4}$  oz. of desiccant was theoretically sufficient to combine with all residual moisture. Packages were stored at 140°F. (RH10%) and 100°F. (RH95%). The moisture content of the product before packaging was 0.25 - 0.3% by analysis. After storage for 2 weeks at 140°F. with desiccant the moisture

was 0.1 percent. (Moisture was analyzed according to AOAC Method, Chapter: Cereal Foods: Wheat Flour: Method: Distillation with Benzene).

In the third experiment, the product was packaged in three different packaging materials with desiccant and stored at 140°F. (RH10%) and 100°F. (RH95%). A  $\frac{1}{4}$  oz. desiccant bag was used for every 3 oz. of dry vinegar.

### Results and Discussion

Table 1 describes the gradual changes in dry vinegar when stored in mason jars at varying temperatures (72°F. - 140°F.). The dry vinegar deteriorated progressively as the temperature was raised over 100°F. whereas below 100°F. the product only had a tendency to cake.

Table 2 and Figures 1, 2, 3 and 4 show the changes in dry vinegar in flexible packages with and without desiccant and stored at 140°F. (10%RH) and 100°F. (95%RH). The product retained the original free-flowing characteristic for 2 months when packaged with desiccant even at 140°F. and 10% RH. Conversely, dry vinegar packaged without desiccant became a brownish slurry in 1 week at 140°F. and in 2 months at 100°F. This shows that in-package desiccation is an effective means in preserving the original properties.

Table 3 and Figures 1, 2, 3 and 4 show the changes which occurred in dry vinegar when packaged in various packaging materials using a desiccant bag and stored at 100°F. (95%RH) and 140°F. (10%RH). Three flexible packaging materials of different laminations were selected for this test. No changes were noted during storage at 140°F. and 100°F. for 2 months when packaged in .0005 inch polyester/.00035 inch foil/.003 inch polypropylene. When dry vinegar was packaged in 25# paper/0.0035 inch foil/0.001 inch polyethylene it became a brownish slurry with a pungent acetic acid odor in 2 months at 100°F. This was also true for dry vinegar packaged in 0.0005 inch polyester/0.00035 inch foil/0.001 inch polyethylene.

### Conclusion

The results of this study confirm the occurrence of chemical reactions in dry vinegar when the temperature of storage is 100°F. or higher. The exact nature of the chemical reactions which take place is not known; however, it is evident that the residual moisture is responsible for irreversible changes. Furthermore, the type of packaging materials is a factor in moisture transfer and subsequent chemical reactions. The use of in-package desiccant and moisture-proof packaging provides an effective means of preventing deterioration in dry vinegar.

### References

1. Oser. U.S. Patent 1,520,366 (1924).
2. Thaddeus C. Kamciek and Kenneth T. Farrell. U.S. Patent 2,696,441, Oct 1953, Dry Imitation Vinegar and Process of Making the Same.
3. Burt, Thomas S. Product Improvement Test of Vinegar Dry Synthetic, USATECOM Project No. 7-EG-925-000-001.

Table 1. Dry Vinegar Stored at Different Temperatures in Mason Jars

<u>Temperature</u>	<u>1 week</u>	<u>2 weeks</u>	<u>3 weeks</u>	<u>1 month</u>	<u>2 months</u>
72°F.	white free flowing powder	white free flowing pdr	white free flowing pdr	caked	caked
85°F.	white free flowing powder	white free flowing pdr	white free flowing pdr	caked	caked
90°F.	white free flowing powder	white free flowing pdr	white free flowing pdr	caked	caked
100°F.	slight caking	slight caking	caked and brown specks	caked and brown specks	completely brown
110°F.	slight caking	caked	caked and brown specks	caked and brown	completely brown
140°F.	caked and brown	completely brown	completely brown slurry	completely brown slurry	completely brown slurry

Table 2. Dry Vinegar Packed in a Flexible Package\*  
with and without Desiccant\*\*

<u>Temperature &amp; Humidity</u>	<u>2 days</u>	<u>1 week</u>	<u>2 weeks</u>	<u>3 weeks</u>	<u>1 month</u>	<u>2 months</u>
<u>100°F (95%RH)</u> <u>with desiccant</u>	white free flowing powder	white free flowing powder	white free flowing powder	white free flowing powder	white free flowing powder	white free flowing powder
<u>w/o desiccant</u>	caked	caked	caked	caked	caked and brown specks	completely brown
<u>140°F. (10%RH)</u> <u>with desiccant</u>	white free flowing powder	white free flowing powder	white free flowing powder	white free flowing powder	white free flowing powder	white free flowing powder
<u>w/o desiccant</u>	brown	completely brown slurry	completely brown slurry	completely brown slurry	completely brown slurry	completely brown slurry

\* 0.0005 inch polyester/0.00035 inch foil/0.003 inch polypropylene.

\*\* Desiccant (CaO) used at ratio of  $\frac{1}{4}$  oz. to 3 oz. of dry vinegar.

Table 3. Dry Vinegar Packaged in Flexible Packaging Materials\* with Desiccant\*\* added.

<u>Temperature &amp; Humidity</u>	<u>1 month</u>	<u>2 months</u>
A. 140°F. (10%RH)	free flowing powder	free flowing powder
100°F. (95%RH)	free flowing powder	free flowing powder
B. 140°F. (10%RH)	free flowing powder	completely turned brown
100°F (95%RH)	free flowing powder	completely turned brown
C. 140°F (10%RH)	free flowing powder	completely turned brown
100°F (95%RH)	free flowing powder	completely turned brown

\* A - 0.0005 inch polyester/0.00035 inch foil/0.003 inch polypropylene.

B - 25# paper/0.0035 inch foil/0.001 inch polyethylene.

C - 0.0005 inch polyester/0.00035 inch in foil/0.001 in polyethylene.

\*\* Desiccant (CaO) used at a ratio of  $\frac{1}{4}$  oz. to 3 oz. of dry vinegar.



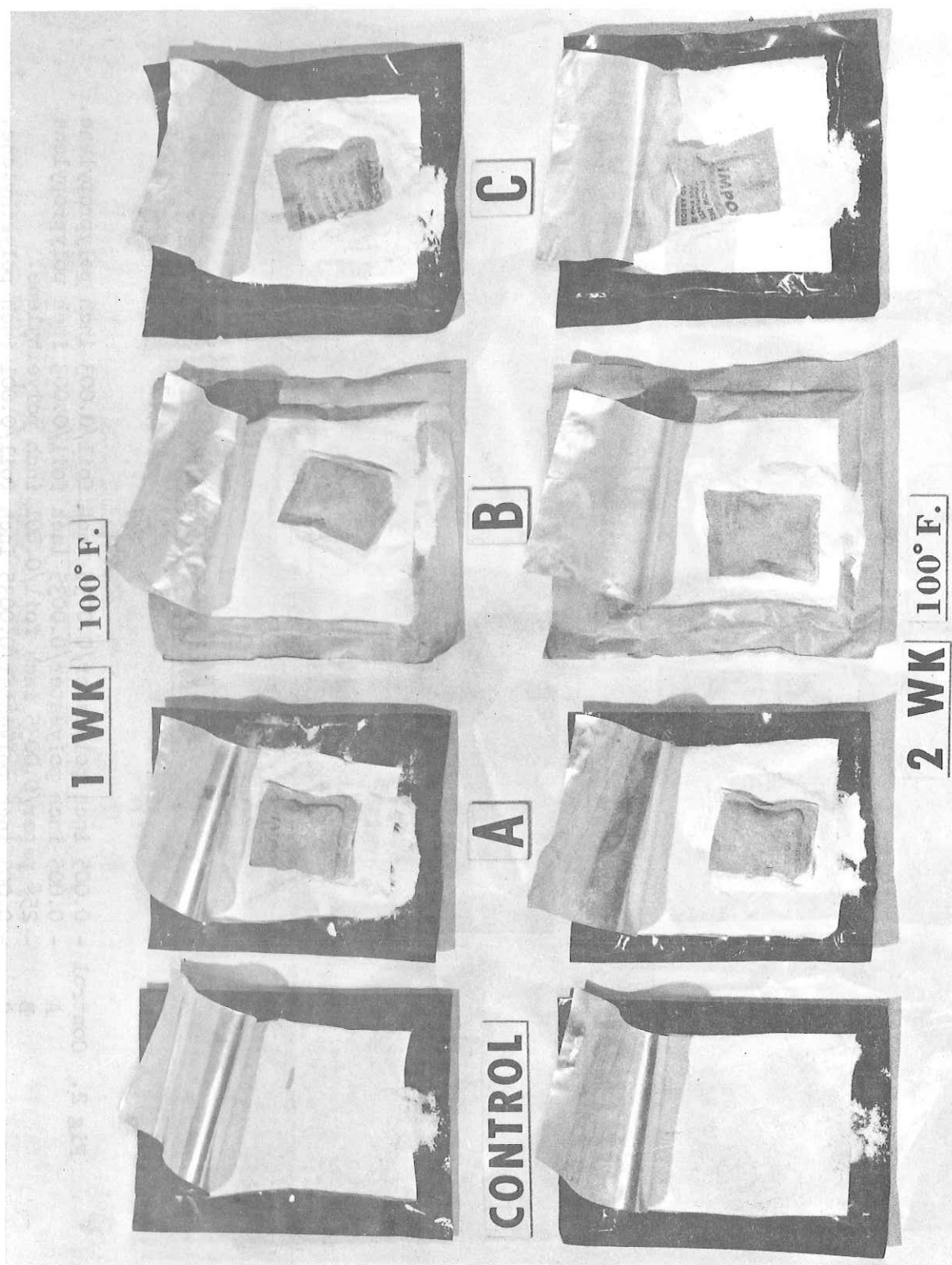


Fig. 1 Control - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
 A - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
 B - 25# paper/0.0035 inch foil/0.001 inch polyethylene.  
 C - 0.005 inch polyester/0.0035 inch foil/0.001 inch polyethylene.

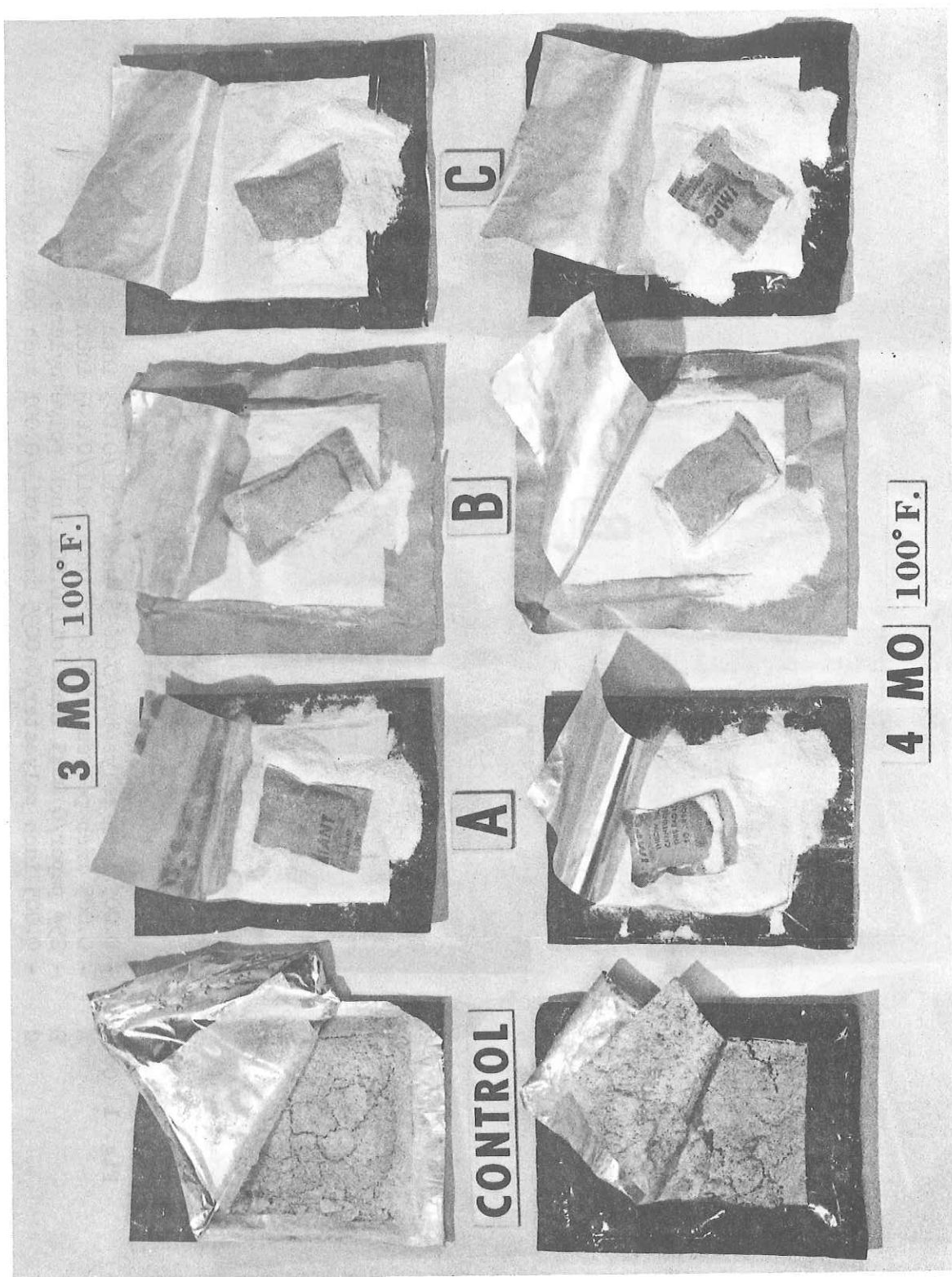


Fig 2. Control - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
 A - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
 B - 25# paper/0.0035 inch foil/0.001 inch polyethylene.  
 C - 0.005 inch polyester/0.0035 inch foil/0.001 inch polyethylene.

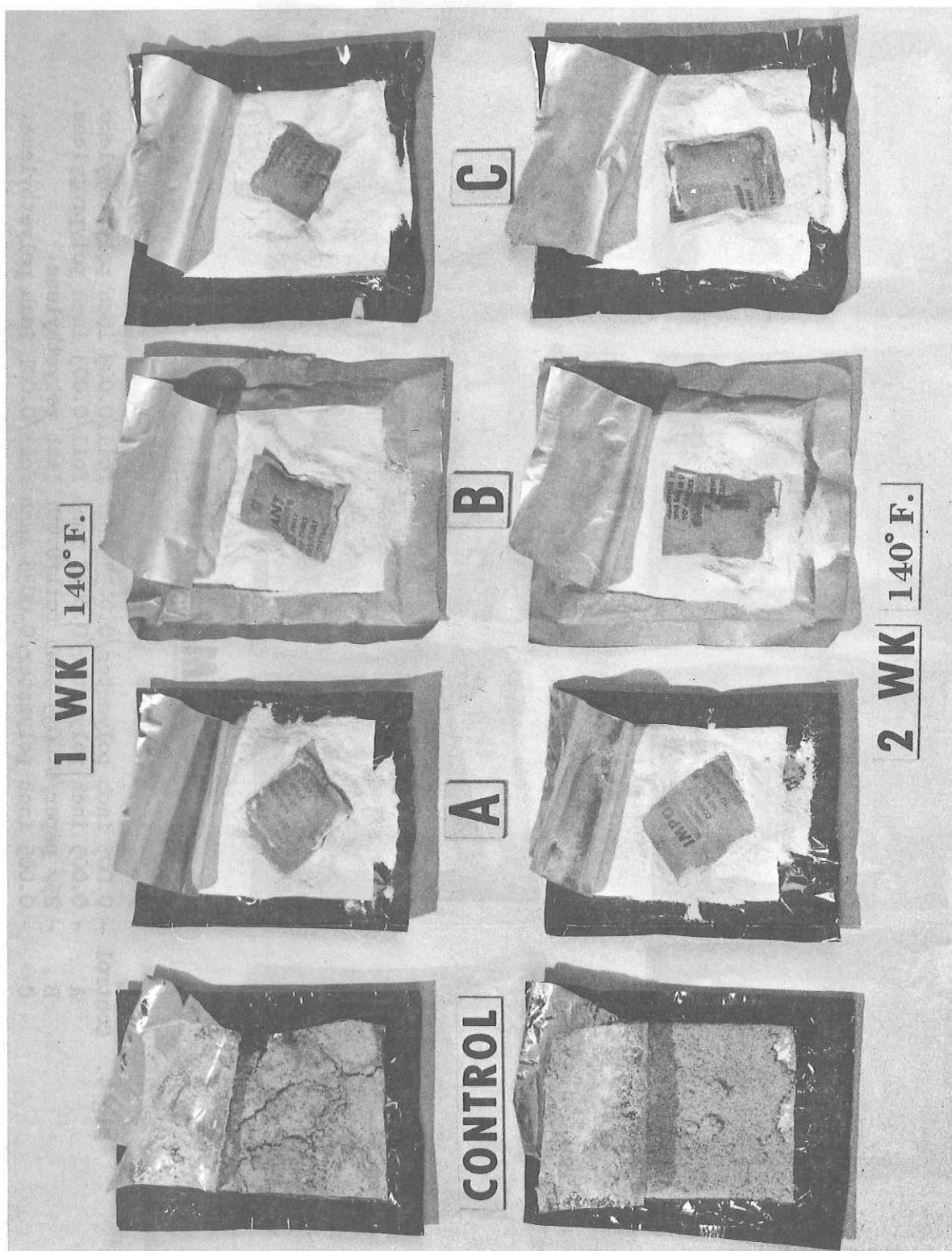
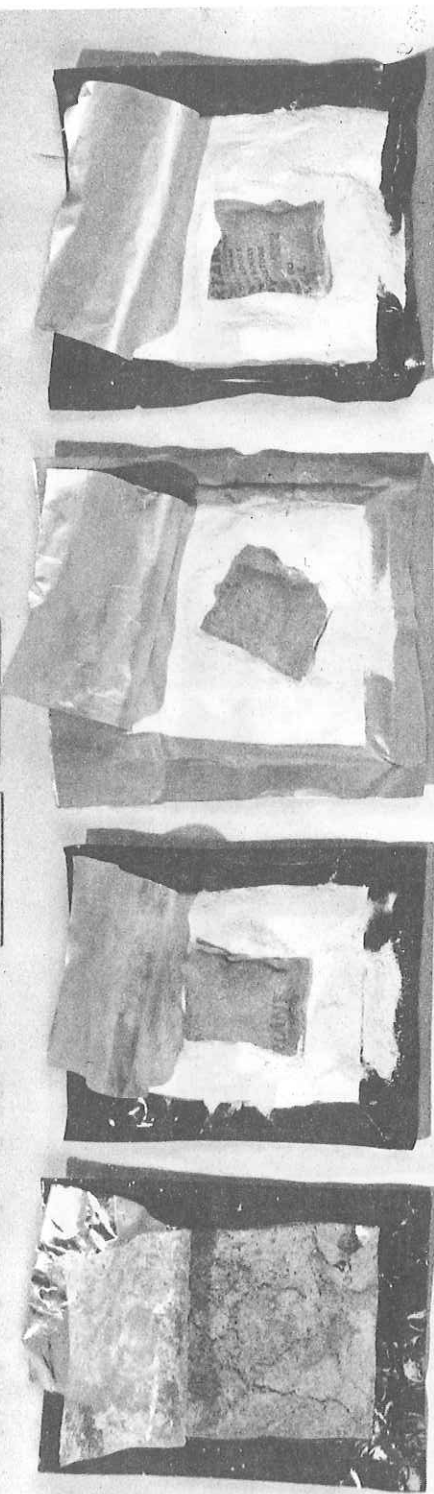


Fig 3. Control - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
 A - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
 B - 25# paper/0.0035 inch foil/0.001 inch polyethylene.  
 C - 0.005 inch polyester/0.0035 inch foil/0.001 polyethylene.

1 MO 140°F.



CONTROL

A

B

C

2 MO 140°F.



Fig 4. Control - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
A - 0.005 inch polyester/0.0035 inch foil/0.003 inch polypropylene.  
B - 25# paper/0.0035 inch foil/0.001 inch polyethylene.  
C - 0.005 inch polyester/0.0035 inch foil/0.001 inch polyethylene.